

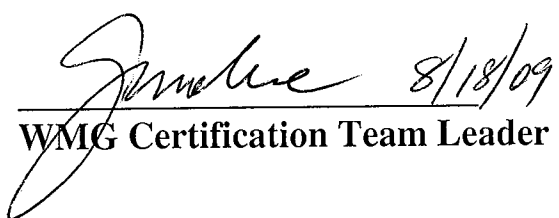
**Bench Top Treatment for Diluting and Neutralizing
Nitric Acid Waste with Magnesium Hydroxide**

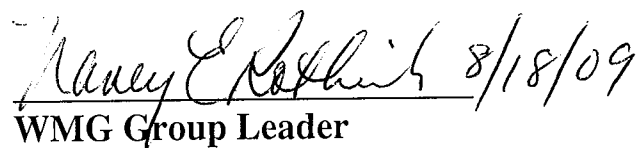
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Submitted by:

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Approved by:

 8/18/09
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Introduction:

A variety of chemical processes are carried out within our group (National Center for Electron Microscopy), which results in the generation of strongly acidic aqueous waste. All of these processes are related to electrochemical-polishing of small transmission electron microscopy samples. These processes are recurring. Mineral acids used in our group include the following:

- Nitric Acid, 70%, 15.8 M

This procedure provides a mechanism for neutralizing these wastes. The nature of the solutions that must be neutralized are described in Pub-3000 and the Chemical Hygiene Safety Plan. Currently, only nitric acid solutions must be neutralized.

Neutralization:

Neutralization must be done prior to transfer to the satellite accumulation area (SAA). In other words, neutralization is the last step in the procedure involving these acids. This means that the acid must be neutralized on the same day that the user is finished with it. Storage of the used acid solution for later neutralization is not permitted!

Documentation:

An accumulation log sheet will be kept for each waste stream detailing the date of generation, volume treated, pH, and treatment date and signature or initial of person performing the treatment. In addition, any additional chemicals present in the waste must be recorded on the log sheet. For example, if the acid solution also contains metals ions or organic compounds, the identities and quantities of these constituents must be recorded on the log sheet. The log sheet will be included with the waste when it is sent to waste management, and a copy of the log sheet will be placed in a binder to act as the local log book. A blank log sheet is included at the end of this procedure.

Training:

All users of this procedure must be trained at least once prior to treating waste. They may be retrained as necessary. Training records must be recorded on the attached log and must be kept for a minimum of three years.

Apparatus:

The waste will be neutralized in a container large enough to contain the solution after it has been diluted to less than or equal to 2 M (note: this is the concentration of the acid, not its normality; in other words, dilute sulfuric acid to 2 M, which will be 4 N). The neutralization container must be in secondary containment large enough to contain the solution if the primary container fails (secondary containment may be the hood itself if the hood has integral secondary containment). A stir bar may be added to the container, and the neutralization may take place on a magnetic stirrer. Alternatively, the container may be gently swirled by hand to mix.

PPE:

A lab coat, long pants, closed toe shoes, long chemical resistant gloves (neoprene, butyl, and Viton are best; heavy nitrile gloves are acceptable), indirectly vented or unvented goggles or face shield and safety glasses.

Engineering Controls:

All treatment will be done in a fume hood.

Disposal:

All waste will be requisitioned for disposal as hazardous waste. The waste will be described as it exists after neutralization based on the predicted reaction products. In other words, a solution of nitric acid that has been neutralized is a mixture of magnesium nitrate and magnesium hydroxide, **not** a mixture of nitric acid and magnesium hydroxide. The presence of suspected trace amounts of any organic or heavy metal constituent must be included in the requisition waste description as appropriate. In cases where the Waste Management Group determines that a requisitioned waste treatment residual is no longer regulated as hazardous, this waste may be discharged to the sanitary sewer with the advice and consent of the Waste Management and Environmental Services Groups.

Acid Neutralization Procedure

- 1) Clear a workspace in the hood to be used to neutralize waste
- 2) Collect necessary lab ware and secondary containment
- 3) Don necessary PPE to protect against chemical splash: lab coat, long chemical resistant gloves (not disposable gloves), long pants, closed toe shoes, and either safety glasses and face shield (preferred) or indirectly vented or unvented goggles (acceptable).
- 4) In a fume hood, dilute acid to less than or equal to 2 M by slowly **pouring acid into water** (If concentration of acid is unknown, pour into 10 volumes of water).
Note: diluting concentrated acids is very exothermic! Keep hood sash between your face and the acid solution.
- 5) Calculate the amount of magnesium hydroxide needed to neutralize the acid (32 g per mole of protons, which includes a 10% excess).
- 6) In a fume hood, add magnesium hydroxide to acid in batches, while stirring the solution to disperse the insoluble magnesium hydroxide. Stirring may be either mechanical (magnetic stirrer) or manual (swirling the mixture by hand).
- 7) Monitor the temperature of the mixture (gloved hand is OK). If the solution becomes hot, wait until the solution is merely warm before proceeding.
- 8) Neutralization is complete when magnesium hydroxide no longer dissolves in the acid solution. However, some magnesium salts, such as magnesium sulfate or magnesium phosphate may precipitate, so this method is not fool-proof.
- 9) Measure the pH of the resulting solution to make sure it is between 6 and 11.
- 10) Transfer the solution to an appropriately labeled container and place in SAA.

**Training Log with List of Authorized Personnel for
Acid Waste Benchtop Neutralization using Magnesium Hydroxide**
(records must be retained for three years)

The following personnel have received on-the-job training in bench top treatment of concentrated nitric acid waste, including

- 1) How the waste was generated,
- 2) Documentation and characterization of waste and how to fill out the Treatment Log
- 3) Safety precautions
- 4) Neutralization procedure
- 5) Requisition the waste (or drain disposal procedures if applicable)
- 6) Emergency response

Name	Signature	Training date	Employee Number

Trained by: _____

Signature: _____

(Keep one copy in Bench Top Treatment Binder)

Treatment: Dilution into water and neutralization with magnesium hydroxide

[illegible]